

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 2740 (1986): Sulphanilic Acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



Indian Standard
SPECIFICATION FOR
SULPHANILIC ACID, TECHNICAL
(*Second Revision*)

UDC 667.281 : 547.551.52



© Copyright 1986

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SULPHANILIC ACID, TECHNICAL

(*Second Revision*)

Dye Intermediates Sectional Committee, PCDC 11

Chairman

DR P. V. SUBRAMANIAM

Representing

Colour-Chem Ltd, Bombay

Members

SHRI D. K. AGGARWAL

Directorate General of Technical Development,
New Delhi

SHRI K. P. DOHARE (*Alternate*)

DR K. APARAJITHAN

Atic Industries Ltd, Bulsar

SHRI A. M. KAPADIA (*Alternate*)

SHRI S. D. BHARDWAJ

Ajanta Chemical Industries, New Delhi

SHRI KAPIL DEV (*Alternate*)

SHRI M. V. DESAI

Atul Products Ltd, Atul

DR J. M. TUREL (*Alternate*)

DR Y. B. DESAI

Universal Dyestuff Industries Ltd, Vadodara

SHRI HIMAT J. DOSHI

Indian Chemical Manufacturers Association, Calcutta

SHRI V. K. MENON

Amar Dye-Chem Ltd, Bombay

SHRI B. M. NAIK

Indian Dyestuff Industries Ltd, Bombay

SHRI D. G. PATWARDHAN

Development Commissioner, Small Scale Industries,
New Delhi

SHRI D. P. SINGH (*Alternate*)

SHRI S. RAJAGOPALAN

Hindustan Ciba-Geigy Ltd, Bombay

SHRI D. K. MURTHY (*Alternate*)

SHRI K. L. RATHI

Sudarshan Chemical Industries Ltd, Pune

DR A. THOMAS (*Alternate*)

DR J. N. SHAH

Sandoz (India) Ltd, Bombay

SHRI K. S. RINDANI (*Alternate*)

SHRI N. R. TALPADE

Hickson & Dadajee Ltd, Bombay

SHRI M. W. SHENDE (*Alternate*)

SHRI S. L. TOSHNIWAL

Zenith Steel Pipes (Chemical Division), Bombay

DR V. V. RAO (*Alternate*)

(*Continued on page 2*)

© Copyright 1986

INDIAN STANDARDS INSTITUTION

This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

(Continued from page 1)

Members

SHRI V. G. UPADHYE
SHRI H. K. VENKATARAMAIAH
SHRI M. S. SAXENA,
Director (P & C)

Representing

Synthofine Chemicals of India Pvt Ltd, Bombay
Hindustan Organic Chemicals Ltd, Rasayani
Director General, ISI (*Ex-officio Member*)

Secretary

SHRI A. KAR
Joint Director (P & C), ISI

Sulphonic Acids Dye Intermediates Subcommittee, PCDC 11 : 2

Convenor

DR S. SIDDHAN

Colour-Chem Ltd, Bombay

Members

SHRI R. V. BUBNA
SHRI RAVI ADUKIA (*Alternate*)
SHRI M. V. DESAI
DR J. M. TUREL (*Alternate*)
DR V. P. KUBBA
SHRI K. L. RATHI
DR A. THOMAS (*Alternate*)
SHRI M. W. SHENDE
SHRI K. P. RANE (*Alternate*)
SHRI H. K. VENKATARAMAIAH

Piramal Rasayan Ltd, Bombay
The Atul Products Ltd, Atul
Indian Dyestuff Industries Ltd, Vadodara
Sudarshan Chemicals Industries Ltd, Pune
Hickson & Dadajee Limited, Bombay
Hindustan Organic Chemicals Ltd, Rasayani

Indian Standard

SPECIFICATION FOR SULPHANILIC ACID, TECHNICAL (*Second Revision*)

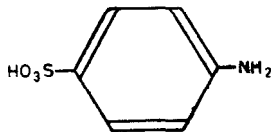
0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 26 February 1986, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first issued in 1964 and revised in 1973. Sulphanilic acid is an important organic intermediate used in organic synthesis and in the production of quinone. It is also widely used in the manufacture of variety of dyes and drugs. The dyestuff industry has made further progress in the recent past and on the basis of the latest development in the industry the Committee decided to undertake the second revision of this standard in order to cater to quality of the product presently available.

0.3 In the present version the requirements of assay and aniline have been revised. The limits of impurities such as orthanilic acid, metanilic acid, and aniline-2, 5-disulphonic acid has been stipulated. Paper chromatographic test method has been introduced to assess these impurities.

0.4 Sulphanilic acid is represented by the following structural formula:



[*p*-amino-benzene-sulphonic acid*; 1-amino-benzene-4-sulphonic acid*]

Molecular mass 173.2

*These names are approved by the International Union of Pure and Applied Chemistry.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for sulphanilic acid, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of grey paste or powder free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR SULPHANILIC ACID, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT*	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Aniline content, percent by mass, <i>Max</i>	0.5	A-2
ii)	Assay content (exclusive of aniline), percent by mass, <i>Min</i>	97.5	A-3
iii)	Matter insoluble in sodium carbonate solution, percent by mass, <i>Max</i>	1.0	A-4
iv)	Ash content, percent by mass, <i>Max</i>	1.0	A-5
v)	Orthanilic acid, metanilic acid and aniline 2, 5-disulphonic acid, percent by mass, <i>Max</i>	1.0	A-6

*On dry basis.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable containers like jute bags or drums conforming to IS : 2552-1979† lined with polyethylene film.

*Rules for rounding off numerical values (revised).

†Specification for steel drums (galvanized and ungalvanized) (second revision).

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer;
- c) Batch number;
- d) Tare, net and gross mass; and
- e) Recognized trade mark; if any.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Preparation of Test Samples — The method of preparing representative samples of the material shall be as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Each individual sample shall be tested for assay.

4.2.2 Test for the determination of the remaining characteristics, namely, aniline content, matter insoluble in sodium carbonate solution, ash and orthanilic acid, metanilic acid and aniline-2'5-disulphonic acid content shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 For Individual Samples — The lot shall be declared as conforming to the requirement of assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 For Composite Samples — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (see 4.2.2), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

*Methods of sampling and test for dye intermediates.

5. TEST METHODS

5.1 Tests shall be carried out according to the method prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A P P E N D I X A

(*Clause 2.2 and Table 1*)

METHODS OF TESTS FOR SULPHANILIC ACID, TECHNICAL

A-1. PREPARED SAMPLE

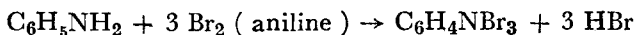
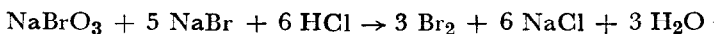
A-1.1 Mix the sample well by rotating the bottle several times and transfer a portion immediately into a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acid or alkaline fumes. Dry the sample at 105°C to constant mass and use this *prepared sample* for tests.

A-2. DETERMINATION OF ANILINE CONTENT

A-2.0 General — Two methods have been prescribed for determination of aniline in sulphanilic acid, namely, bromide bromate titration method and the thin layer chromatographic method. The titration method (**A-2.1**) shall be employed in case of dispute and the chromatographic method (**A-2.2**) shall be employed for routine analysis.

A-2.1 Bromide Bromate Titration Method

A-2.1.0 Outline of the Method — An accurately weighed quantity of the material is dissolved in alkaline solution and aniline is distilled off into dilute hydrochloric acid. The distillate containing the aniline is titrated with previously standardized bromide bromate solution, according to the following reaction:



*Specification for water for general laboratory use (*second revision*).

A-2.1.1 Apparatus**A-2.1.1.1 Distillation flask** — 2-litre capacity.**A-2.1.1.2 Condenser** — of not less than 45 cm length.**A-2.1.2 Reagents****A-2.1.2.1 Sodium hydroxide solution** — approximately 20 percent (*m/v*).**A-2.1.2.2 Concentrated hydrochloric acid** — conforming to IS : 265-1976*.**A-2.1.2.3 Sodium thiosulphate solution** — approximately 0.1 N.

A-2.1.2.4 Standard bromide bromate solution — 0.1 N. Dissolve 10 g of sodium bromide and 2.5 g of sodium bromate in 1 000 ml water. Pipette out 100 ml aliquot from the above solution in a 1 000-ml iodine flask. Add 100 ml of water, 5 g of potassium iodide and shake to dissolve. Then add 15 ml of concentrated hydrochloric acid rapidly and stopper the flask. Shake vigorously. Allow to stand for 5 minutes. Titrate with the standard sodium thiosulphate solution using 2 ml starch solution as an indicator. Disappearance of blue colour indicates end point. Calculate the normality of bromide-bromate solution as follows:

$$\frac{\text{Titre reading} \times \text{Normality of sodium thiosulphate solution}}{100}$$

NOTE — Same solution shall not be used after three months.

A-2.1.2.5 Starch and potassium iodide test paper

A-2.1.3 Procedure — Place 17 g of the *prepared sample* accurately weighed in a 2-litre distillation flask. Add to it 45 ml of sodium hydroxide solution to dissolve and then dilute with water to a volume of 400 ml. Add 3 or 4 glass beads. The solution shall be alkaline to phenolphthalein. Connect the side arm of the distillation flask with the condenser. Distil until free from aniline when tested as follows:

Collect about 50 ml distillate in a 500-ml conical flask containing 50 ml of 1:1 hydrochloric acid solution. Titrate at room temperature with standard bromide-bromate solution. Test the solution by spotting on starch and potassium iodide papers. The end point is marked when an immediate faint blue ring appears which can be obtained repeatedly during a period of 5 minutes without further addition of bromide-bromate solution.

Repeat the process of collecting the distillate in 50 ml lots and titrating as above till not more than 2 to 3 drops of bromide-bromate solution are required.

*Specification for hydrochloric acid (*second revision*).

A-2.1.4 Calculation

$$\text{Aniline content, percent by mass} = \frac{V \times N \times 1.55}{M}$$

where

V = volume in millilitres of standard bromide-bromate solution used,

N = normality of bromide-bromate solution, and

M = mass in grams of the material taken for the test.

A-2.2 Chromatographic Method

A-2.2.0 Outline of the Method — Aniline content is estimated by ascending type thin layer chromatographic separation on silica gel plate.

A-2.2.1 Apparatus

A-2.2.1.1 Chromatographic chamber — $22 \times 10 \times 22$ cm.

A-2.2.1.2 Micropipette — 10 μ l capacity.

A-2.2.1.3 Thin layer chromatographic glass plate — 20×20 cm.

A-2.2.1.4 Adjustable thin plate applicator

A-2.2.1.5 Chromatographic spray bottle

A-2.2.1.6 Oven — with a thermo regulator.

A-2.2.2 Regents

A-2.2.2.1 Solution of aniline — 0.01 percent in methanol on 100 percent basis.

A-2.2.2.2 Eluent — Benzene : Chloroform 95:5 (v/v).

A-2.2.2.3 Sodium nitrite solution — 1 N.

A-2.2.2.4 Hydrochloric acid — 1 N.

A-2.2.2.5 Spray reagent — 0.1 percent H-Acid solution in 1 N caustic soda solution.

A-2.2.2.6 Silica-gel G — Neutral.

A-2.2.3 Procedure

A-2.2.3.1 Preparation of chromatographic plate — Mix 50 g of neutral silica-gel with 85 to 90 ml of water for three minutes. Then by means of the applicator, coat the plate with silica gel layer of thickness not exceeding 0.25 mm and place it in the oven maintained at 110°C for 90

minutes. By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

A-2.2.3.2 Preparation of the sample solution — Homogenize the sample for a few minutes and weigh by means of an weighing bottle 1.0 g of the material and transfer to a 50-ml volumetric flask. Dissolve in methanol with the help of ammonia solution on (1:1) and make up the solution to the mark by adding additional quantity of methanol.

A-2.2.3.3 Chromatographic Separation — Pipette out 10 μ l of the sample solution (A-2.2.3.2) and 10 μ l of the referee solution (A-2.2.2.1) in the form of a uniform line on the plate at a distance of 3 cm from the bottom edge. After complete evaporation of the solvent place the plate in the chromatographic chamber and allow the eluent (A-2.2.2.2) in the chamber to migrate for about 120 minutes. When it reaches the upper line of the plate remove the plate and let it dry in air for at least 30 minutes. Dry the plate completely, diazotize in nitrous fumes [1 N NaNO₂ + 1 N HCl, (1:2) (v/v)] and couple with reagent (A-2.2.2.5) by spraying. Let the plate stand for at least 15 minutes. A zone of colour characteristic to aniline appears as under:

Substance	Zone	R _f	Colour
Aniline	I	0.30	Pink

A-2.2.4 Evaluation — By visual comparison the spot corresponding to aniline shall have equal or less intensity than that of the reference sample corresponding to 0.5 percent of aniline.

A-3. DETERMINATION OF AMINE CONTENT (EXCLUSIVE OF ANILINE)

A-3.1 Determine the total amine content in accordance with the method prescribed in 12.1 of IS : 5299-1969*.

A-3.2 Reporting — The total amine content determined shall be corrected for the amount of aniline (determined under A-2) and expressed as amine content.

A-3.3 Calculation

$$\frac{\text{Amine content (excluding aniline), percent by mass on dry basis (molecular/mass 173.2)}}{A - \frac{173.2 \times B}{93.12}}$$

where

A = total amine content (inclusive of amine), percent by mass, on molecular mass 173.2; and

B = aniline content, percent by mass on molecular mass 93.12.

*Methods of sampling and tests for dye intermediates.

A-4. MATTER INSOLUBLE IN SODIUM CARBONATE SOLUTION

A-4.1 Determine matter insoluble in sodium carbonate solution in accordance with **10.2** of IS : 5299-1969*.

A-5. ASH CONTENT

A-5.1 Determine ash content in accordance with **11.1** of IS : 5299-1969*.

A-6. DETERMINATION OF ORTHANILIC ACID, METANILIC ACID, SULPHANILIC ACID AND ANILINE — 2, 5-DISULPHONIC ACID

A-6.0 Outline of the Method — The contents of impurities in sulphanilic acid, technical is determined by paper chromatography.

A-6.1 Apparatus

A-6.1.1 *Paper Chromatographic Chamber*

A-6.1.2 *Micropipette* — of 10 μ l capacity.

A-6.2 Reagents

A-6.2.1 *Chromatographic Paper* — Whatman No. 4 in the form of a strip of size 25 $\times$ 16 cm.

A-6.2.2 *Eluent* — Concentrated ammonia/water/*n*-butanol (10:10:90).

A-6.2.3 *Ammonia-Water Mixture* — 1:1 (*v/v*).

A-6.2.4 *Spray Solution* — 0.5 percent aqueous solution of American Base (N-1-Naphthylethylenediamine dihydrochloride).

A-6.3 Procedure

A-6.3.1 Weigh accurately 0.5 g of the material and dissolve in 100 ml of a mixture of concentrated ammonia and water (in the proportion of 1:1 volume). Similarly prepare solutions of the reference sample of 0.002 5 percent 0.005 percent, 0.01 percent and 0.015 percent concentrations. Take the chromatographic paper and mark a start line at a distance of 3 cm from any one end of the paper. Spot 10 microlitre each of the test solution and reference solutions along the start line at a distance of 3 cm from one another.

A-6.3.2 Saturate the chromatographic chamber with the eluent by placing a trough containing the eluent at the bottom of the closed chamber. Suspend the chromatographic strip in the chamber in such

*Methods of sampling and tests for dye intermediates.

a manner that the spotted end of the paper strip just dips into the trough containing the eluent. The eluent will travel upwards along the strip. Allow it to develop in this manner for 2 h during which time the solvent front travels approximately 20 cm past the start line. Remove the strip from the chamber, dry it in air.

Diazotize with nitrous fumes and spray with spray solution (A-6.2.4). Following zones of characteristic colours appear:

<i>Components</i>	<i>Zone</i>	<i>Rf Value</i>	<i>Colour</i>
Aniline-2, 5-disulphonic acid	I	0.02	Pink
Sulphanilic acid	II	0.15	Red
Metanilic acid	III	0.19	Pink
Orthanilic acid	AI	0.38	Orange

A-6.4 Reporting

A-6.4.1 Report impurity content as that which is nearest in intensity to the reference sample. In case, the colour intensity does not come in the range of the standard spots of impurity, repeat the whole procedure using suitable concentration of various impurities.



INDIAN STANDARDS INSTITUTION

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 331 0131 331 1375

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:

Telephone

*Western : Manakalaya, E9 MIDC, Marol Andheri (East) 6 32 92 95
BOMBAY 400093

†Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, 36 24 99
Maniktola, CALCUTTA 700054

Northern : SCO 445-446, Sector 35-C { 2 18 43
CHANDIGARH 160036 { 3 16 41

Southern : C. I. T. Campus, MADRAS 600113 { 41 24 42
{ 41 25 19
{ 41 29 16

Branch Offices:

‘Pushpak’, Nurmohamed Shaikh Marg, Khanpur { 2 63 48
AHMADABAD 380001 { 2 63 49

‘F’ Block, Unity Bldg, Narasimharaja Square, 22 48 05
BANGALORE 560002

Gangotri Complex, 5th Floor, Bhadbhada Road, 6 67 16
T. T. Nagar, BHOPAL 462003

Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002 5 36 27

53/5 Ward No. 29, R. G. Barua Road, 5th Byelane, —
GUWAHATI 781003

5-8-56C L. N. Gupta Marg, HYDERABAD 500001 22 10 83

R14 Yudhister Marg, C Scheme, JAIPUR 302005 { 6 34 71
{ 6 98 32

117/418 B Sarvodaya Nagar, KANPUR 208005 { 21 68 76
{ 21 82 92

Patliputra Industrial Estate, PATNA 800013 6 23 05

Hantex Bldg (2nd Floor), Rly Station Road, 52 27
TRIVANDRUM 695001

Inspection Office (With Sale Point):

Institution of Engineers (India) Building, 1332 Shivaji Nagar, 5 24 35
PUNE 411005

*Sales Office in Bombay is at Novelty Chambers, Grant Road, 89 65 28
BOMBAY 400007

†Sales Office in Calcutta is at 5 Chowringhee Approach, 27 68 00
P.O. Princep Street, CALCUTTA 700072